RESEARCH

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Discovery

Nypa fruticans (NIPA) starch filled with zinc oxide nanoparticles as corrosion inhibitor in steel

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Metal oxide nanoparticles have attracted tremendous interest as corrosion inhibitor due to their high surface area on metal surfaces. It is well known that zinc oxide nanoparticles (ZnONPs) have higher reactivity on basic and acidic solution. This article presents a method of synthesizing zinc oxide nanoparticles using *Nypa fruticans (NIPA)* starch and zinc oxide nanoparticles' effect on corrosion inhibition in steel. Steel substrates were prepared and immersed in 0.5 M NaCl aqueous solution at 30 ^{0}C. The corrosion inhibition efficiency of 25% ZnONPs was determined by weight loss measurement, atomic force microscopy (AFM), scanning electron microscopy (SEM) and electron impedance spectroscopy (EIS) methods. Weight loss measurement was calculated and evaluated within 7 days and suggested that ZnONPs are effective as corrosion inhibitor. The surface morphology of the steel in the absence and presence of ZnONPs was investigated using Atomic Force Microscopy (AFM) and Scanning Electron Microscopy (SEM) techniques and the outcomes indicated a formation of a protective layer over the steel substrate with the ZnONPs. The Electron Impedance Spectroscopy (EIS) showed that charge transfer controls the corrosion process. The results and analyses of this work indicated that a zinc oxide nanoparticle is effective as corrosion inhibitor in steel.

INTRODUCTION

Nyfa fruticans is commonly used as roof (leaves) in the province of Northern Samar, Philippines. The uses of this palm especially its fruits remained unnoticed to the people. Northern Samar is an island and it implies the increase of corrosion rates on steel like appliances, automobiles and buildings. Since the presence of Sodium chloride mixed with Oxygen or water molecules increases the reaction of metal alloys to form corrosion. Nowadays, people live comfortably with machines, automobiles and other devices that make life easier. These devices are mostly made of metals. But corrosion has the major impact in people's economic and everyday life. Rust and corrosion are costing the world industry with a lot of money every year. Coatings like paints and oils are efficient ways for corrosion protection of metals. Zinc oxide is one of those paint ingredients. Nanotechnology utmost improves the quality of life. The study of minute particles with size ranging from one to one hundred nanometers (1-100nm) is called nanotechnology. When ZnO is reduced to nanoscale, it shows unique properties in comparison to its bulk counterpart. These unique properties of ZnONPs are due to enhanced surface area, which allows for increased interaction of nanoparticles.

In view of the complex community concern about corrosion, this study therefore aims to determine if the Nipa palm starch filled with Zinc oxide (ZnO) nanoparticles are effective as corrosion inhibitor in steel. This study entails us to accomplish the goals of testing the synthesized zinc oxide nanoparticles from *Nypa fruticans (NIPA)* starch as corrosion inhibitor in steel using weight loss measurement, atomic force microscopy (AFM), scanning electron microscopy (SEM) and electron impedance spectroscopy (EIS).

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PROCEDURAL METHODS

Preparation of biosynthesized Zinc Oxide Nanoparticles from Nypa fruticans (nipa) starch

A study entitled "Synthesis, Characterization and Application of Zinc Oxide Nanoparticles in Biotechnology" by Subhankar Paul and Deependra Kumar Ban published by Int'l Journal of Advances in Chemical Engg. & Biological Sciences in 2014 focused on synthesizing zinc oxide nanoparticles in biological system using zinc nitrate and starch as the stabilizing agent. Zinc oxide nanoparticles (ZnONps) were prepared by using zinc nitrate ($Zn(NO)_2$) and sodium hydroxides precursors and starch as a stabilizing agent. *Nypa fruticans* starch about 0.1 g was dissolved in 500 mL of lukewarm distilled water. Zinc nitrate, 14.874 g (0.1 mol), was added in the above solution, and then followed by constant stirring for 1 hour using magnetic stirrer to completely dissolve the zinc nitrate. After complete dissolution of zinc nitrate, 0.2 mol of sodium hydroxide solution was added drop by drop under constant stirring. The reaction was allowed to proceed for 2 h. After the completion of reaction, the solution was kept overnight and the supernatant solution was discarded carefully. Rest of the solution was centrifuged at 10,000 g for 10 min and the supernatant was discarded. Thus, the nanoparticles were obtained and washed thrice using distilled water. Washing was carried out to remove the by-products and the excessive starch bound with the nanoparticles. After washing, the

nanoparticles were dried at 80^{O}C overnight. During drying, complete conversion of $Zn(OH)_2$ into ZnONps has been taken place.

Preparation of Metal Substrates for Corrosion Inhibition Test

Mild steel sheets were used in this study. Metal sheets were purchased from Betty Enterprises (construction market) located at Brgy. Bangkerohan, Catarman, Northern Samar and the steel sheets were mechanically cut into coupons with dimensions of 1.5 cm x 1.5 cm and were degreased in ethanol. Surface polishing was done using different grades of silicon carbide electro-coated water-proof abrasive papers then cleaned with acetone, air-dried, weighed and immersed immediately in the test solutions with and without 25% zinc oxide nanoparticles in 0.5 M Sodium chloride (NaCl).

Weight Loss Measurement

Pre-weighed steel substrates were immersed in 100 mL of the blank/inhibitor solutions for 7 days. The weight loss of each coupon has been determined at every 24 h interval by retrieving the substrates from the solution, cleaning with acetone and then reweighing. The mass loss for each sample was evaluated by dividing its weight loss by the surface area of the substrates. Series of weight loss studies were carried out on pre-weighed steel substrates which were immersed in 100 mL capacity beakers containing 100 mL of the test solutions which were maintained at 30^{o}C temperature. All weights were obtained using analytical balance. By denoting the initial weight and the final weights of the coupons as W_1 and W_2 respectively, corrosion rates, inhibition effectiveness (inhibition efficiency ($\mathcal{E}_{WL}$%)), and degree of surface coverage (Θ) were calculated as follows:

$C_R = (W1 - W2)/At$ Equation (1)

$\mathcal{E}_{WL} = 100\,x\,\left(\frac{C_{Rblank} - C_{Rinh}}{C_{Rblank}}\right)$ Equation (2)

$\Theta = 0.01\,(\mathcal{E}_{WL})$ Equation (3)

Where Rb and Ri are the corrosion rates in the absence and presence of the inhibitor and A is the surface area (cm^2) of the metal specimens and $\boldsymbol{t}$ is the temperature of the test solutions. This formulas were taken from the published journal of Journal of Materials and Environmental Sciences entitled "Elephant Grass Biomass Extract as Corrosion Inhibitor for Mild Steel in Acidic Medium" researched by E.B. Ituen, A.O. James, and O. Akaranta.

Atomic Force Microscopy (AFM)

Atomic Force Microscopy was used to characterize the morphology before and after the electrochemical reaction of starch/ZnONps on steel substrates. Atomic Force Microscopy (AFM) imaging was done under ambient conditions with a PicoSPM II (PicoPlus, Molecular Imaging – Agilent Technologies) in the Magnetic ACMode (MAC Mode) using a magnetic field to drive a magnetically coated cantilever in the top-down configuration. Type II MAC Levers with a spring constant of 2.8 nN/M with about 10 nm tip radius was used for all scans.Surface studies is similar to the published journal titled "Studies on the Inhibition of Mild Steel Corrosion in Hydrochloric Acid Solution by Atenolol Drug" by G. Karthik and M. Sundaravadivelu on Egyptian Journal of Petroleum.

Scanning Electron Microscopy (SEM)

The scanning electron microscopy (SEM) VEGA3TESCAN model was used to study the morphology of the corroded surface in the presence and absence of zinc oxide nanoparticles (ZnONPs) for the immersion of 43 d at room temperature. The SEM images were taken from that portion of the specimen where better information was expected.Methods for the Scanning electron microscopy was taken from the published journal titled "Studies on the Inhibition of Mild Steel Corrosion in Hydrochloric Acid Solution by Atenolol Drug" by G. Karthik and M. Sundaravadivelu on Egyptian Journal of Petroleum.

Electron Impedance Spectroscopy (EIS)

For EIS measurement, methods were taken from the published study submitted in NRCP Research Journal entitled "Electrochemical Deposition of PVK/MWNTs CPN Nanocomposite Films for Anti-corrosion Application" researched by Dr. Karina Milagros Cui-Lim, Christina A. Binag and Rigoberto C. Advincula. Electrochemical impedance spectroscopy (EIS) measurements were performed using the Autolab PGSTAT12 potentiostat to investigate the change of electrochemical system and interface within pure starch, Zinc oxide, zinc oxide nanoparticles and test solution without zinc oxide nanoparticles. Corrosion tests were carried out on electrodes cut from sheets of stainless steel. EIS in the absence of redox species was used to estimate the corrosion-protection performance of ZnONPs coatings against aqueous 0.5 M NaCl solution. The impedance data were recorded after 43 days of immersion of stainless steel substrates in corrosive medium of salt water (0.5 M $NaCl_{aq}$). An Autolab PGSTAT12 potentiostat equipped with Frequency Response Analyzer (FRA) Eco Chemie B.V. software (Utrecht, Netherlands) was utilized, along with a three (3) electrode system of an aqueous Ag/AgCl (3.5 M NaCl) reference electrode (RE), a platinum wire as auxiliary electrode (AE), and the coated steel samples/ITO glass as a working electrode (WE). The EIS measurements for the ZnONPs, starch, ZnO and without ZnONPs were performed under open circuit potential in an ac frequency range from 100,000 to 0.01 Hz with an excitation signal of 5 mV sinusoidal perturbations. All electrochemical experiments were carried out at room temperature. The FRA instrument was connected to a computer and used in conjunction with Boukamp software. The Boukamp software, provided by FRA Research is used for EIS analysis for fitting in an equivalent circuit.

RESULTS AND DISCUSSION

Effectivity of zinc oxide nanoparticles (ZnONps) as corrosion inhibitor has gained positive result as presented in its anti-corrosion studies through weight loss measurement, atomic force spectroscopy, scanning electron microscopy and electron impedance spectroscopy. The results of this study were further illustrated through tables and graphs in the succeeding sections.

Weight Loss Measurement

Values of corrosion rates, percentage inhibition efficiency and degree of surface coverage were calculated from weight loss method of solution without zinc oxide nanoparticles and solution with 25% zinc oxide nanoparticles at 30^{o}C after 24 h within 7 days of immersion. They were summarized at Table 1. It was observed that zinc oxide nanoparticles inhibit the corrosion of the steel in 0.5 M NaCl. The inhibition efficiency was 68.25% and degree of surface coverage resulted to 0.6825. The degree of surface coverage is the surface covered by the corrosion in the experiment.

As shown in Figure 1, the corrosion rate for metal substrate soaked in 0.5 M NaCl with 25% ZnONPs in 7 days has 1.04 x $10^{-5}$and the maximum corrosion rate was 4.44 x 10^{-5}. The metal substrate soaked in 0.5 M NaCl without ZnONPs resulted values from 3.70 x 10^{-5} to 7.7 x 10^{-5} which indicated that corrosion rates on metal substrate in 0.5 M

Table 1 Corrosion rate, inhibition efficiency and degree of surface coverage for steel corrosion in 0.5 M NaCl at 30 °C temperature

DAY	SAMPLES	CORROSION RATES ($gcm^{-2}h^{-1}$)	INHIBITION EFFICIENCY (%)	DEGREE OF SURFACE COVERAGE ((θ))
	w/ 25% ZnONps	1.04×10^{-5}	74	0.0074
1	w/out ZnONps	4×10^{-5}		
2	w/ 25% ZnONps	1.48×10^{-5}	60	0.0060
	w/out ZnONps	3.70×10^{-5}		
3	w/ 25% ZnONps	1.04×10^{-5}	75.81	0.0076
	w/out ZnONps	4.30×10^{-5}		
4	w/ 25% ZnONps	4.44×10^{-5}	42.34	0.0042
	w/out ZnONps	7.7×10^{-5}		
5	w/ 25% ZnONps	1.93×10^{-5}	67.45	0.0067
	w/out ZnONps	5.93×10^{-5}		
	w/ 25% ZnONps	2.67×10^{-5}	59.92	0.0060
6	w/out ZnONps	6.67×10^{-5}		
7	w/ 25% ZnONps	2.07×10^{-5}	53.59	0.0054
	w/out ZnONps	6.52×10^{-5}		
AVERAGE TOTAL:			68.25	0.0068

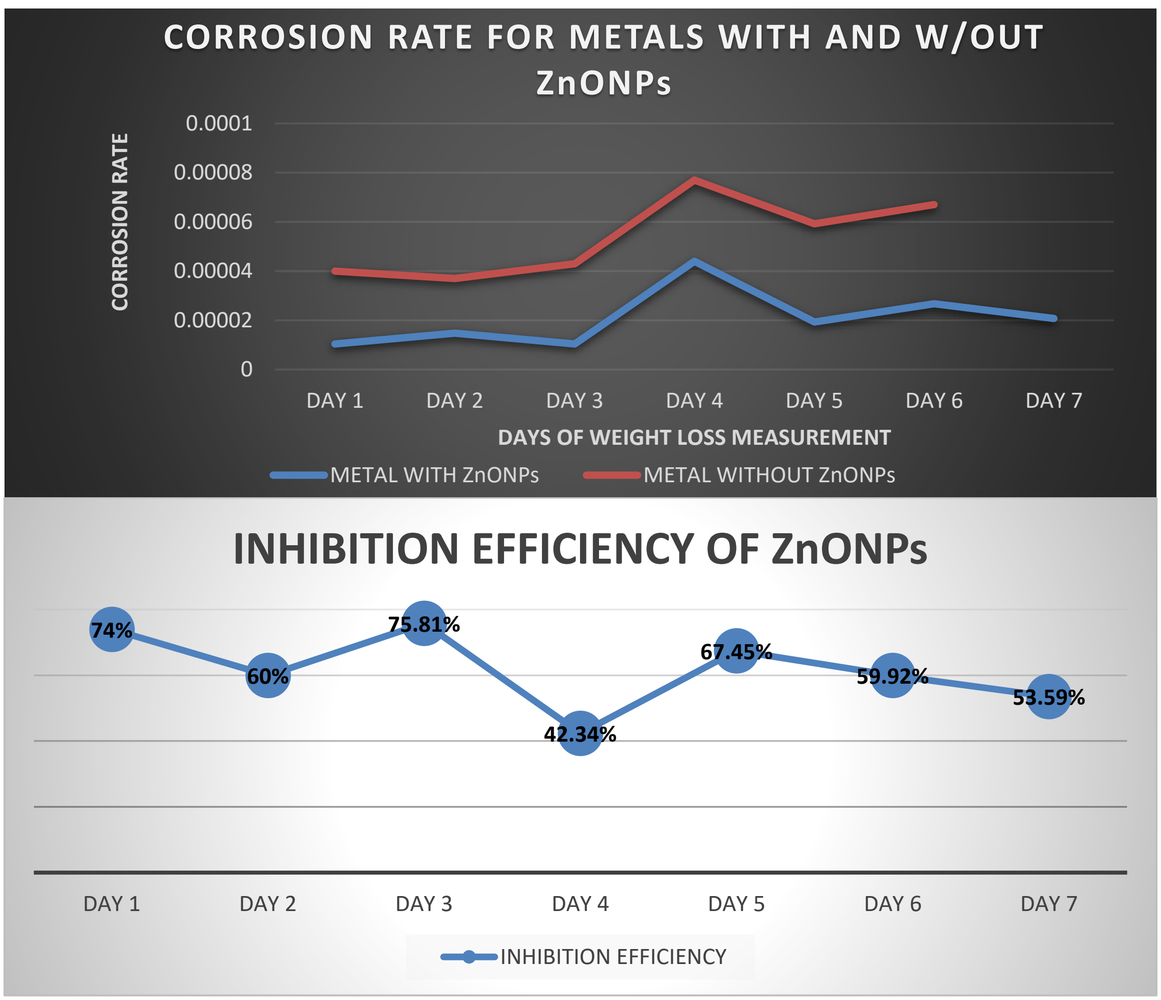
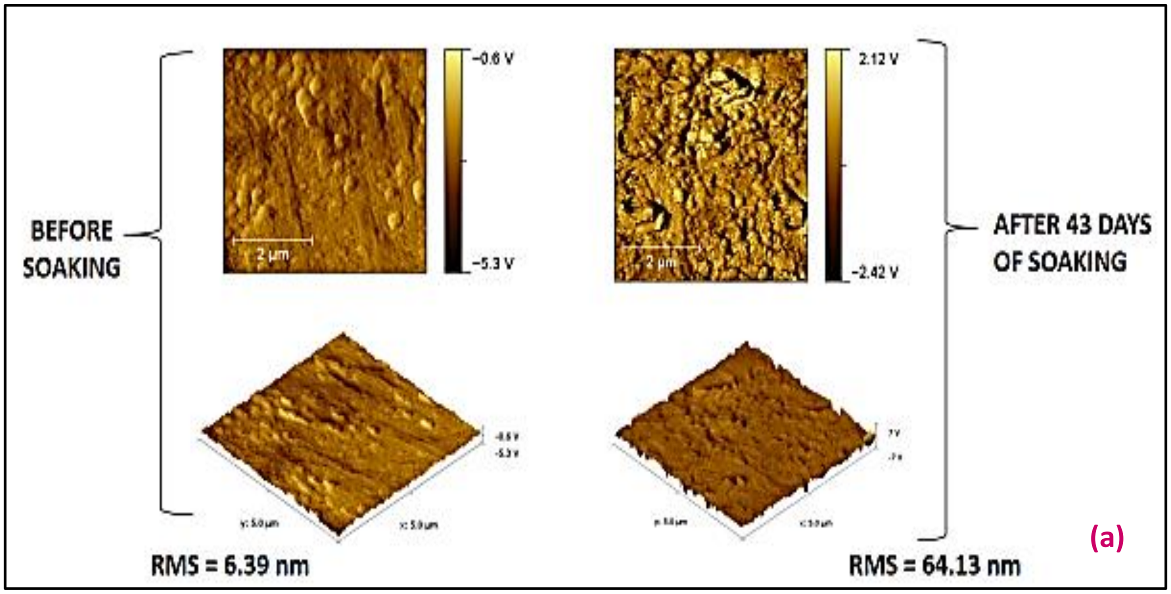


Figure 1 Graph of corrosion rates and inhibition efficiency of metal substrates in 0.5 M NaCl with and without ZnONPs



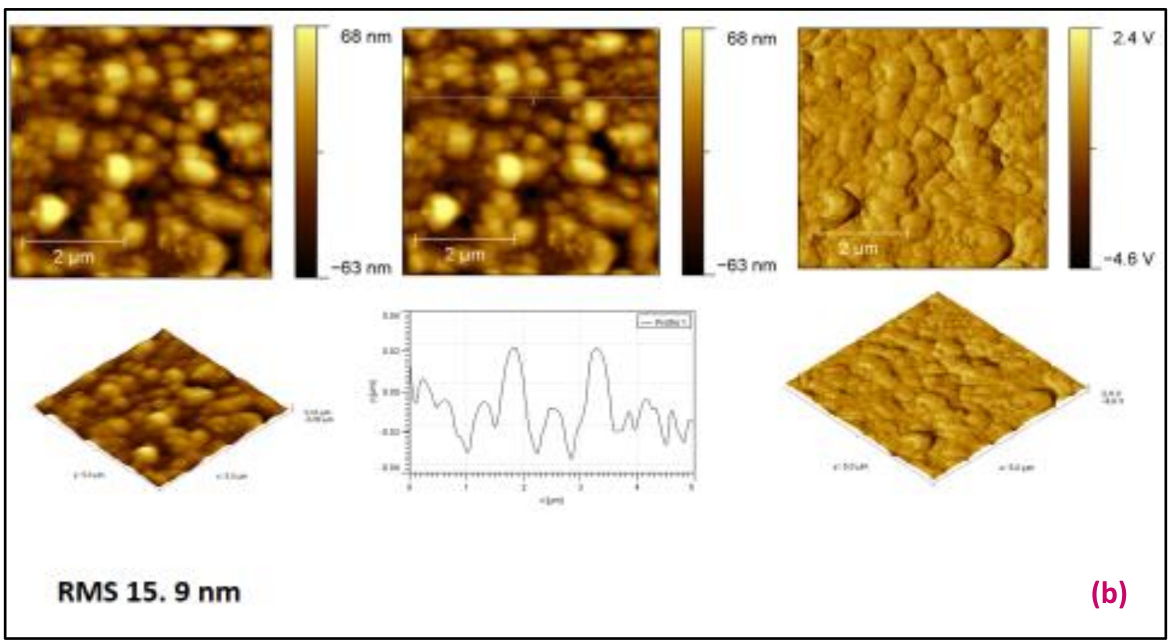


Figure 2 (a) AFM phase images of bare steel without ZnONPs before and after soaking (b) AFM images of steel in ZnONPs solution after soaking for 43 days

Table 3 The differences of RMS measurement in AFM images

Test Solutions	RMS Before Soaking	RMS After 43 d of Soaking	RMS Difference
Without ZnONPs	6.39 nm	64.13 nm	57.74 nm
With Starch	6.39 nm	48.50 nm	42.11 nm
With zinc oxide	6.39 nm	32.50 nm	26.11 nm
With ZnONPs	6.39 nm	15.9 nm	9.51 nm

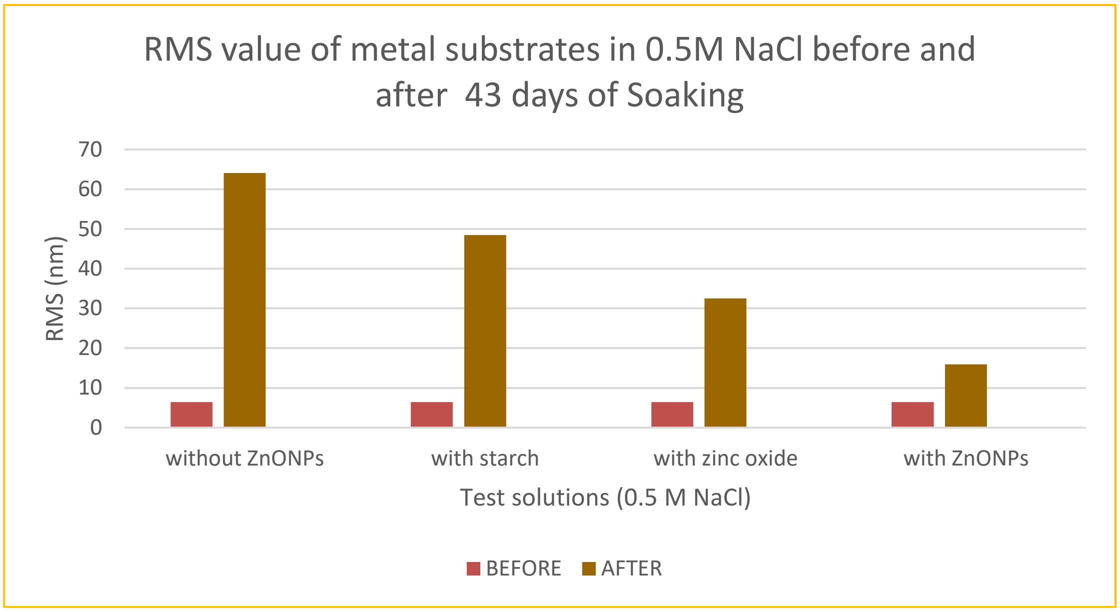


Figure 3 Graph of the RMS value for the AFM Analysis

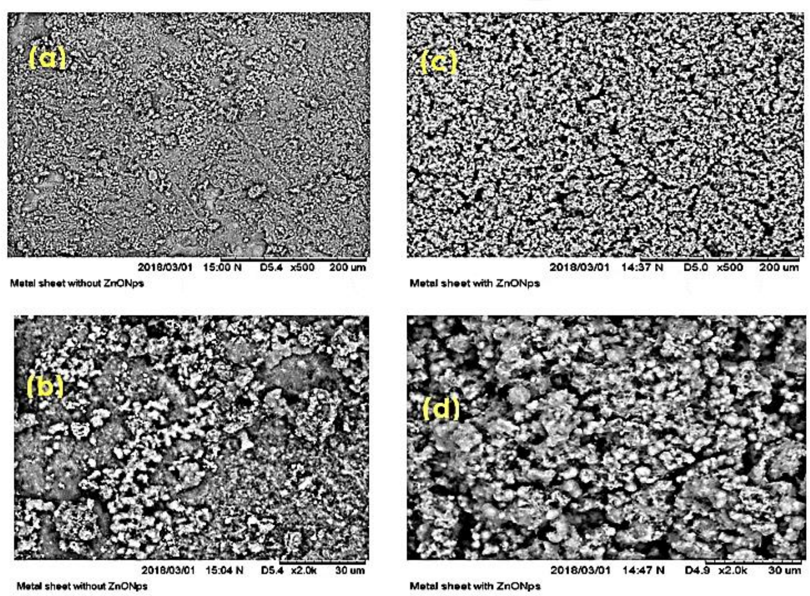


Figure 4 SEM images of metal specimens (a) steel without ZnONPs at x500 magnification, (b) steel without ZnONps at x2000 magnification, (c) steel with ZnONPs at x500 magnification and (d) steel with ZnONPs at x2000 magnification.

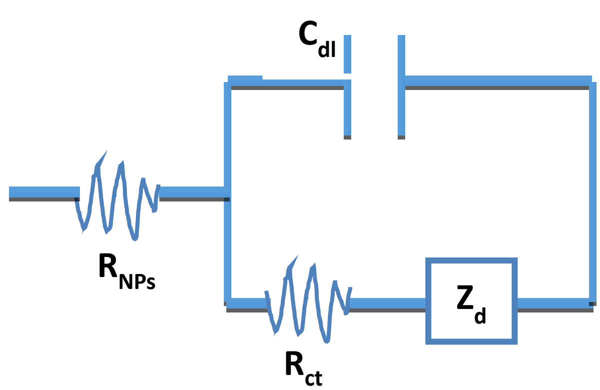


Figure 5 Randles circuit used in interpretation of EIS data
where, $\mathbf{R_{NPs}}$ = electrolyte resistance; $\mathbf{C_{dl}}$ = capacitance of Zinc Oxide Nanoparticles
$\mathbf{R_{ct}}$ = charge transfer resistance of the metalsubstrate to corrosion

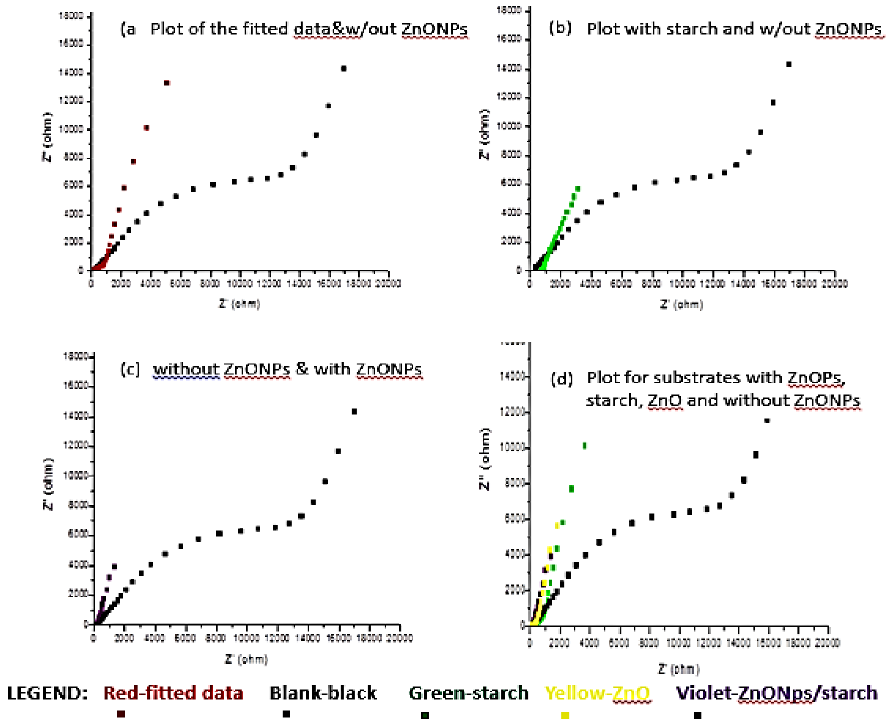


Figure 6 Nyquist plot which is made by plotting the imaginary impedance component (Z") against the real component (Z') at each excitation frequency

NaCl without ZnONPs are higher than the metal substrate soaked in the test solution with ZnONPs. As seen in the figure, the metal with 25% ZnONPs has lower corrosion rate than the metal without the ZnONPs.

Atomic Force Microscopy

The result as presented in Figure 2 (a) was seen that corrosion occurred after 43 days of immersion. From the roughness of the metal which was measured as 6.39 nm before soaking in the 0.5 M $NaCl_{aq}$ solution, it increased to 64.13 nm without the zinc oxide nanoparticles. As expected, there was certain damage in the steel due to the absence of corrosion inhibitor/ZnONPs. Figure 2(b) evaluated that with the presence of ZnONPs in the test solution with the metal substrate, the roughness of the corrosion only increased from 6.39 nm up to 15.9 nm. It showed that with the presence of ZnONPs, corrosion inhibition is higher.

From the initial roughness measurement of surface which is 6.39 nm before soaking, the steel that has the lowest increase of roughness measurement was the one immersed in the 0.5 M NaCl with zinc oxide nanoparticles rather than the steels immersed without ZnONPs, with starch and zinc oxide. This result proved that ZnONPs are effective as corrosion inhibitor in steel as seen on the two dimensional AFM images.

Figure 3 evaluated the differences of the metal substrates roughness measurements. As seen in Figure 11, the metal substrate soaked in the test solution without the zinc oxide nanoparticles has the highest measure of roughness indicating that corrosion is immediately increasing compared to the metal substrates with starch, zinc oxide and ZnONPs solution. The metal soaked with the ZnONPs resulted to the lowest roughness measurement indicating that ZnONPs is effective as inhibitor on corrosion processes in steel.

Scanning Electron Microscopy (SEM)

Scanning electron microscope images evaluated the conditions of metal surface in contact with 0.5 M NaCl solution in the absence and presence of zinc oxide nanoparticles as inhibitor. A surface analysis was carried out using SEM immediately after the tests of corrosion. As shown in Figure 12 (c-d) below, surface corrosion of the steel decreased due to the presence of zinc oxide nanoparticles. Steel without zinc oxide nanoparticles as shown in figure 12(a-b) revealed that there is severe damage, clear pits and cavities on the surface of the steel than the steel with the presence of ZnONPs. There were fewer cracks and pits observed in the inhibited surface. It conforms that the steel or metal surface is fully covered with the ZnONPs/starch molecules and a protective coating that acts as inhibitor was formed. Scanning electron microscopy aided in the surface morphology of the metal substrates immersed in the test solutions with and without ZnONPs by its clear and reliable source of imaging.

Electron Impedance Spectroscopy (EIS)

From the circuit elements, the charge-transfer resistance (R_{ct}) of the zinc oxide nanoparticles on the steel substrate is a measure of the ZnONPs' ability to act as barrier to the corrosion process, the R_{NPs} represents the extent of ionic conduction through a coating in an electrolyte environment and is widely used as a criterion for assessing the extent of corrosion protection that can be derived from nanoparticle coatings, while the double layer capacitance (C_{dl}) of coating used in place of the coating capacitance by taking into account the surface heterogeneity and diffusion processes also provides information on the extent of water uptake and the stability of the nanoparticles. The equivalent electrical circuit that consists the active electrolyte resistance which is the zinc oxide nanoparticles in series with the parallel combination of the double

layer capacitance is called the Randles circuit shown on Figure 5. It interpreted the impedance spectra and one of the simplest model describing the presence of electrochemical interface.

Figure 6 shows the experimental results obtained from EIS measurements for the corrosion of steel in the absence and presence of starch, Zinc oxide and Zinc oxide nanoparticles at room temperature. The impedance spectra for steel in 0.5 M NaCl solution without ZnONPs, starch, ZnO and with 25% concentration of zinc oxide nanoparticles are presented as Nyquist plots in Figure 14. Red Nyquist plot indicated the fitted data of the impedance spectra. The black Nyquist plot evaluated the impedance spectra of the metal soaked in 0.5 M NaCl solution without ZnONPs. Green Nyquist plot evaluated the impedance spectra of the steel soaked in starch solution. The yellow Nyquist plot identified the impedance spectra of the metal soaked in zinc oxide solution and the violet Nyquist plot determined the impedance spectra of the metal soaked in 0.5 M NaCl with ZnONPs solution. Clearly, the impedance spectra exhibit a large capacitive loop at high frequencies followed by a small inductive loop at low frequency values. The capacitive loop indicates that the corrosion of steel is mainly controlled by a charge transfer process, and usually related to the charge transfer of the corrosion process and double layer behaviour. On the other hand, the inductive loop may be attributed to the relaxation process obtained by adsorption of inhibitor on the electrode surface. The diameter of the capacitive loop in the presence of inhibitor is bigger than in the absence of inhibitor (blank solution) and increases with the starch, ZnO and ZnONPs. This indicates that the impedance of inhibited substrate increases with the presence of ZnONPs.

CONCLUSION

Zinc oxide nanoparticles are effective as corrosion inhibitor in steel based on the results obtained from weight loss measurement, atomic force microscopy, scanning electron microscopy and electron impedance spectroscopy. As proven from recent researches involving nanoparticles, zinc oxide nanoparticles have the inhibition performance during corrosion processes.

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